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Indian Standard

SPECIFICATION FOR ZINC OXIDE AND ZINC SALTS FOR ELECTROPLATING

(*First Revision*)

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Indian Standard

SPECIFICATION FOR ZINC OXIDE AND ZINC SALTS FOR ELECTROPLATING

(First Revision)

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Indian Standard

SPECIFICATION FOR ZINC OXIDE AND ZINC SALTS FOR ELECTROPLATING

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 17 October 1977, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

0.2 For the electro-deposition of zinc, the electrolytic baths used are generally classified as being either of the alkaline or the acid type. The alkaline baths are usually based on the double cyanide of the metal. Zinc oxide is used to prepare the double cyanide bath and to restore balance when the alkaline baths are impoverished in metal. The acid baths, on the other hand, are based on zinc sulphate or zinc chloride or often both, and contain a proportion of other salts also.

0.3 This standard was first published in 1967 under the title ' Specification for zinc oxide for electroplating '. However, keeping the requirements of the electroplating industry in view, the Sectional Committee responsible for the preparation of the standard decided to revise it and make it more comprehensive by incorporating the requirements of zinc sulphate and zinc cyanide which were prescribed in IS : 2290-1962* and IS : 3027-1964† respectively. In this revision requirements have also been prescribed for zinc sulphate monohydrate and zinc chloride which were not covered in any of these specifications earlier. As a consequence of the publication of this comprehensive revised standard IS : 2290-1962* and IS : 3027-1964† are superseded.

0.4 In the formulation of this standard, due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from BS 2656 : 1972 ' Zinc anodes, zinc oxide and zinc salts for electroplating ', issued by the British Standards Institution.

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*Specification for zinc sulphate for electroplating

†Specification for zinc cyanide for electroplating.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for zinc oxide and the following zinc salts for electroplating:

a) Zinc cyanide,
b) Zinc sulphate (hepta and monohydrate), and
c) Zinc chloride.

2. REQUIREMENTS

2.1 Description — Zinc oxide and zinc salts shall conform to the following description:

Name of Material	*Description*
Zinc oxide	Dry, white powder, free from extraneous matter and corresponding essentially to the formula ZnO.
Zinc cyanide	White or greyish white powder, free from extraneous matter and corresponding essentially to the formula Zn (CN)2.
Zinc sulphate	Solid crystals or white crystalline powder, free from extraneous matter and corresponding essentially to the formula $ZnSO_4.7H_2O$ or $ZnSO_4.H_2O$.
Zinc chloride	Either in the form of very hygroscopic and deliquescent white to light coloured, fused solid or granules; or solution of relative density as agreed to between the purchaser and the supplier. In either case it shall be free from carbon or metallic particles, dirt or any other visible impurity.

NOTE — In case of zinc chloride supplied in the form of a solution which is not perfectly clear and shows flocculent precipitate due to the presence of zinc oxychloride, an aliquot of 200 ml of 10 percent (*m/v*) solution shall become entirely clear on addition of 2 ml of dilute hydrochloric acid (1 : 1).

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*Rules for rounding off numerical values (*revised*).

2.2 Zinc oxide and the zinc salts shall also comply with the requirements given in Table 1, when tested in accordance with the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 8 of the table.

NOTE — In the case of zinc chloride being in the form of a solution, it shall first be dried and fused at 270°C and then only tested in accordance with the methods prescribed in Appendix A.

3. SAFETY PRECAUTIONS IN HANDLING ZINC CYANIDE

3.1 Zinc cyanide is extremely poisonous and shall only be handled with extreme care. Useful information on this subject is given in Appendix B.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in air-tight, galvanized steel containers, having an opening of a suitable size, so that the contents are prevented from taking up moisture from air, or from getting contaminated by contact with metals or other foreign matter when small quantities are withdrawn from time to time.

4.1.1 Zinc chloride solution shall be packed in carboys made of glass or other suitable inert material as agreed to between the purchaser and the supplier.

4.2 Marking — The containers shall be marked with the following:

a) Name, form and net mass of the material;
b) Name of the manufacturer or his recognized trade-mark, if any; and
c) Relative density and total solid content in the case of zinc chloride solution.

4.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The method of drawing representative samples of material and the criteria for conformity shall be as prescribed in Appendix C.

TABLE 1 REQUIREMENTS FOR ZINC OXIDE AND ZINC SALTS FOR ELECTROPLATING

(*Clauses* 2.2, A-4.3.3, A-6.3.3, A-8.3.1.1, C-5.1 *and* C-5.2)

Sl No.	Characteristic	Zinc Oxide	Zinc Cyanide	Zinc Sulphate		Zinc Chloride	Ref to Method of Test
				Hepta Hydrate	Mono Hydrate		
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Zinc oxide (as ZnO), percent by mass, *Min*	99·0	—	—	—	—	A-2
ii)	Zinc (as Zn), percent by mass, *Min*	—	54·0	22·5	35·0	45·5	A-2
iii)	Cyanide (as CN), percent by mass, *Min*	—	40·0	—	—	—	A-3
iv)	Lead (as Pb), percent by mass, *Max*	0·015	0·005	0·01	0·015	0·01	A-4
v)	Cadmium (as Cd), percent by mass, *Max*	0·01	0·01	0·01	0·015	0·01	A-5
vi)	Mercury (as Hg), percent by mass, *Max*	0·003	0·002	0·001	0·001	—	A-6
vii)	Copper (as Cu), percent by mass, *Max*	—	0·05	0·02	0·03	—	A-7
viii)	Iron (as Fe), percent by mass, *Max*	0·007	0·05	0·02	0·03	0·002	A-8
ix)	Matter insoluble in cyanide, percent by mass, *Max*	0·005	—	—	—	—	A-9
x)	Matter insoluble in water, percent by mass, *Max*	—	—	0·1	0·1	—	A-10

APPENDIX A

(*Clause* 2.2)

ANALYSIS OF ZINC OXIDE AND ZINC SALTS FOR ELECTROPLATING

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF ZINC AND ZINC OXIDE

A-2.0 General — Zinc is estimated by titrating the solution of the material against standard EDTA solution. Zinc oxide content, where required, is calculated from the result by multiplying by the conversion factor 1·245.

A-2.1 Reagents

A-2.1.1 *Concentrated Hydrochloric Acid* — approximately 36 percent (*m/v*) (*see* IS : 265-1976†).

A-2.1.2 *Triethanolamine Solution* — 10 percent (*v/v*).

A-2.1.3 *Buffer Solution* — Prepare by adding 54 g of ammonium chloride to 350 ml of ammonia solution [approximately 20 percent (*m/m*)] and dilute to 1 litre.

A-2.1.4 *Potassium Cyanide Solution* — 20 percent (*m/v*).

A-2.1.5 *Indicator Solution* — Prepare by dissolving 0·4 g of Eriochrome Black T and 3 g of hydroxyl-ammonium chloride in 100 ml of rectified spirit (*see* IS : 323-1959‡).

A-2.1.6 *Hydrogen Cyanide Detection Papers* — Prepare as required by dipping strips of filter paper in a mixture of equal volumes of solutions A and B, prepared as follows:

Solution A	0·1 percent (*m/v*) o-tolidine acetate. Dissolve 0·64 g of o-tolidine in 5 ml of 50 percent (*m/v*) glacial acetic acid and dilute to 1 000 ml with water.
Solution B	0·3 percent (*m/v*) copper acetate solution in water

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*Specification for water for general laboratory use (*second revision*).

†Specification for hydrochloric acid (*second revision*).

‡Specification for rectified spirit (*revised*).

A-2.1.7 *Formaldehyde Solution* — 10 percent (*v/v*). Dilute 100 ml of formaldehyde to 1 000 ml with water.

A-2.1.8 *Standard Disodium Ethylenediamine Tetra-Acetate (EDTA) Solution* — Dissolve 18·6 g of disodium ethylenediamine tetra-acetate dihydrate in water and make up the volume to 1 litre in a volumetric flask. Then weigh accurately 2·000 g of reagent grade zinc metal and dissolve in 10 ml of dilute hydrochloric acid (1 : 1). Transfer to a 250-ml volumetric flask and dilute with water to the mark. Transfer 25 ml of this solution to a 400-ml beaker and proceed as in **A-2.2.1.1** to confirm the end point. Note the volume (V_1) of EDTA solution consumed.

A-2.2 Procedure

A-2.2.1 *Zinc Oxide, Zinc Sulphate and Zinc Chloride* — Weigh accurately about 5 g of the material, transfer to a 400-ml beaker and dissolve in 100 ml of water containing 15 ml (1 ml in the case of sulphate and chloride) of concentrated hydrochloric acid. Transfer to a 250-ml volumetric flask and dilute with water to the mark. Reserve the solution for subsequent tests.

NOTE — In the case of zinc chloride *see* Note under **2.2**.

A-2.2.1.1 Transfer 50-ml aliquot to a 400-ml beaker and neutralize to litmus with triethanolamine solution. Add 10 ml of buffer solution and dilute to 200 ml. Add 10 ml of potassium cyanide solution, 1 ml of Eriochrome Black T indicator solution, 20 ml of formaldehyde solution and titrate with standard EDTA solution to the appearance of a blue colour. Add a further 10 ml of the formaldehyde solution and continue the titration to the removal of the last trace of a red colour in the solution. Confirm that the end point is permanent by the addition of a further 5 ml of the formaldehyde solution. Note the volume (V_2) of EDTA solution consumed.

A-2.2.1.2 *Calculation*

$$\text{Zinc (as Zn), percent by mass} = \frac{100\,V_2}{V_1 M}$$

where

V_2 = volume in ml of EDTA solution used in **A-2.2.1.1**,

V_1 = volume in ml of EDTA solution used in **A-2.1.8**, and

M = mass in g of the material in the aliquot taken for the test.

A-2.2.1.3 Calculate the zinc oxide content, percent by mass by multiplying the above result by the factor 1·245.

A-2.2.2 *Zinc Cyanide* — Weigh accurately about 5 g of the material and transfer to a 400-ml beaker with 15 ml of water. Transfer to an efficient fume chamber. Cautiously add 50 ml of concentrated hydrochloric acid, cover the beaker with a watch-glass and boil until all hydrogen cyanide is expelled. Test the vapour from the beaker with hydrogen cyanide detection paper. Wash down the watch glass and sides of the beaker with water and evaporate to a syrupy consistency. Cool, dissolve in 100 ml of water containing 1 ml of concentrated hydrochloric acid tranfer to a 250-ml volumetric flask and dilute to the mark. Transfer 50-ml aliquot of this solution to a 400-ml beaker and continue as in **A-2.2.1.1** and **A-2.2.1.2**. Reserve the rest of the solution for subsequent tests.

A-3. DETERMINATION OF CYANIDE IN ZINC CYANIDE

A-3.0 General — Cyanide is determined by direct titration of zinc cyanide solution with standard silver nitrate solution in the presence of potassium iodide.

A-3.1 Reagents

A-3.1.1 *Sodium Hydroxide Solution* — 10 percent (*m*/*v*).

A-3.1.2 *Potassium Iodide Solution* — 10 percent (*m*/*v*).

A-3.1.3 *Standard Silver Nitrate Solution* — 0·1 N approximately.

A-3.2 Procedure — Weigh accurately about 0·5 g of the material, dissolve in 5 ml of sodium hydroxide solution, and dilute with 100 ml of water. Add 2 ml of potassium iodide solution and titrate with standard silver nitrate solution with constant stirring until the first trace of a permanent yellow turbidity is seen in the liquid by transverse illumination against a black background.

A-3.3 Calculation

$$\text{Cyanide (as CN), percent by mass} = \frac{5{\cdot}2\, V N}{M}$$

where

V = volume in ml of standard silver nitrate solution used,
N = normality of standard silver nitrate solution, and
M = mass in g of the material taken for the test.

A-4. DETERMINATION OF LEAD

A-4.0 General — Lead is determined by matching the colour produced by sodium sulphide with the colour produced with standard lead solution.

A-4.1 Apparatus

A-4.1.1 *Nessler Cylinders* — 100 ml capacity (*see* IS : 4161-1967*).

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*Specification for Nessler cylinders.

A-4.2 Reagents

A-4.2.1 *Concentrated Hydrochloric Acid* — same as in **A-2.1.1.**

A-4.2.2 *Hydrogen Cyanide Detection Papers* — same as in **A-2.1.6.**

A-4.2.3 *Concentrated Nitric Acid* — *See* IS : 264-1976*.

A-4.2.4 *Copper Solution* — Dissolve 0·25 g of electrolytic copper in 10 ml of dilute nitric acid (1 : 1) in a beaker and boil off most of the nitric acid. Dilute, if necessary, to get a clear solution.

A-4.2.5 *Citric Acid* — Crystalline solid.

A-4.2.6 *Ammonium Hydroxide Solution* — 20 percent (*m*/*m*).

A-4.2.7 *Sodium Hydrosulphide Solution* — Saturate sodium hydroxide solution, 4 percent (*m*/*v*), with hydrogen sulphide gas, allow to stand for a few days and filter.

A-4.2.8 *Concentrated Sulphuric Acid* — *See* IS : 266-1961†.

A-4.2.9 *Ammonium Nitrate* — Crystalline solid.

A-4.2.10 *Rectified Spirit* — 95 percent (*v*/*v*) (*see* IS : 323-1959‡).

A-4.2.11 *Ammonium Acetate Solution* — 43 percent (*m*/*v*) made just acid to litmus with acetic acid.

A-4.2.12 *Ammoniacal Cyanide Solution* — Mix 100 ml of potassium cyanide solution (10 percent *m*/*v*) with a mixture of 40 ml of ammonium hydroxide solution and 60 ml of water. Filter the mixed solution, if necessary.

A-4.2.13 *Sodium Sulphide Solution* — 10 percent (*m*/*v*) solution of sodium sulphide ($Na_2S.9H_2O$).

A-4.2.14 *Sodium Cyanide Solution* — 10 percent (*m*/*v*).

A-4.2.15 *Standard Lead Solution* — Dissolve 1·598 g of lead nitrate in water, add 5 ml of concentrated nitric acid, and dilute to 1 000 ml. Again dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution contains 0·01 mg of lead (as Pb).

A-4.3 Procedure

A-4.3.1 *Zinc Oxide, Zinc Cyanide and Zinc Chloride* — Weigh accurately 20·0 g of the material and transfer to a 400-ml low-form beaker with 50 ml of water. Place in a well-ventilated fume cupboard and add

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*Specification for nitric acid (*second revision*).

†Specification for sulphuric acid (*revised*).

‡Specification for rectified spirit (*revised*).

carefully 70 ml of concentrated hydrochloric acid (in the case of zinc cyanide boil the solution after addition of concentrated hydrochloric acid until hydrogen cyanide is expelled. Test the vapour from the beaker with hydrogen cyanide test prepared and finally cool). Then add 5 ml of concentrated nitric acid and the prepared copper solution. Add 20 g of citric acid and when solution is complete, cautiously neutralize with ammonium hydroxide solution adding excess until the precipitate first formed is completely re-dissolved. Add 7 ml of sodium hydrosulphide solution and filter the precipitate thus formed without washing. Discard the filtrate.

A-4.3.1.1 Place the filter paper, alongwith the precipitate, back into the precipitation beaker, add 10 ml of water, 15 ml of concentrated nitric acid, 10 ml of concentrated sulphuric acid and evaporate until dense fumes are evolved. If charring occurs, add a minimum quantity of ammonium nitrate to effect complete decomposition and continue fuming for 10 minutes. Cool and add 50 ml of water, pouring it around the sides of the beaker with constant swirling to dissolve the salts completely. Heat the solution to boil and then cool. Add 35 ml of rectified spirit, mix, cover and allow to stand for 4 hours.

A-4.3.1.2 Filter the precipitate through a sintered glass crucible (G. No. 4) which has been previously washed with rectified spirit (30 percent *v/v*). Measure 2·5 ml of 30 percent (*v/v*) rectified spirit and add it drop by drop into the precipitation beaker, swirling to cover the area originally occupied by the precipitate. Pour slowly into the crucible and drain. Repeat the operation with a further 2·5 ml of 30 percent (*v/v*) rectfied spirit. Discard the filtrate and washings. Wash out the filtering flask thoroughly with water and reconnect to the filter.

A-4.3.1.3 Dissolve the precipitate in the sintered glass crucible by alternate washing with hot ammonium acetate solution and hot water using in all 10 ml of ammonium acetate solution and collecting the solution and washings in the filter flask. Cool and transfer the contents to a 500-ml volumetric flask and dilute with water to the mark.

A-4.3.1.4 Transfer 5 ml of the above solution to a 100-ml Nessler cylinder and add 10 ml of ammoniacal cyanide solution. Dilute to the mark and add two drops of sodium sulphide solution. Carry out a control test, proceeding exactly as with the material using standard lead solution as follows:

a) For zinc oxide	3 ml
b) For zinc cyanide	1 ml
c) For zinc chloride	2 ml

Finally mix the contents in the Nessler cylinders and compare the colours produced.

A-4.3.2 *Zinc Sulphate* — Weigh accurately 0·200 g of the material and dissolve in about 20 ml of water in a 100-ml Nessler cylinder. Add 30 ml of sodium cyanide solution and dilute to the mark. In the second Nessler cylinder take 2 ml of standard lead solution for heptahydrate and 3 ml for monohydrate and proceed as in **A-4.3.1.4**. Mix the contents in the Nessler cylinders and compare the colours produced.

A-4.3.3 The limits prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-5. DETERMINATION OF CADMIUM

A-5.0 General — Cadmium is determined by measuring the optical density of the colour of cadmium dithizone complex in a photoelectric absorptiometer at the wave length of 510 nm.

A-5.1 Apparatus

A-5.1.1 *Photoelectric Absorptiometer*

A-5.2 Reagents

A-5.2.1 *Sodium Hydroxide Solution* — 2 percent (*m*/*v*).

A-5.2.2 *Dilute Hydrochloric Acid* — approximately 1 N.

A-5.2.3 *Purified Carbon Tetrachloride* — Reflux 300 ml of carbon tetrachloride with 100 ml of 5 percent (*m*/*v*) solution of sodium hydroxide for two and a half hours, separate and wash well with water, dry over calcium chloride for 1 to 2 hours and re-distill from calcium oxide.

A-5.2.4 *Dithizone Solution* — Dissolve 0·02 g of dithizone in 100 ml of purified carbon tetrachloride, shake in a separating funnel with 100 ml of water and 10 ml of ammonium hydroxide (20 percent *m*/*m*). Reject the carbon tetrachloride layer. Wash the acquous layer with 5 ml of carbon tetrachloride and reject the carbon tetrachloride layer. Acidify the acquous layer with concentrated hydrochloric acid (*see* IS : 265-1976*), cool and extract with three 30-ml portions of carbon tetrachloride. Combine these three extracts, wash three times with water and finally adjust the volume with carbon tetrachloride to 100 ml.

Note — The solution shall be freshly prepared before use.

A-5.2.5 *Sodium Potassium Tartrate Solution* — Measure 45 ml of sodium hydroxide solution (10 percent *m*/*v*) and 5 ml of sodium potassium tartrate solution (20 percent *m*/*v*) into a 100-ml separating funnel. Extract with successive 5-ml portions of dithizone solution, rejecting each

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*Specification for hydrochloric acid (*second revision*).

extract until the solvent layer is not deeper than pale yellow in colour. Finally wash the aqueous layer with carbon tetrachloride and discard the washings.

NOTE — A few drops of chloroform may be added as stabilizer.

A-5.2.6 *Standard Cadmium Solution* — Dissolve 2·04 g of cadmium chloride in water, add 50 ml of concentrated hydrochloric acid, and dilute with water to 1 000 ml. Dilute 10 ml of this solution again to 1 000 ml. One millilitre of the diluted solution contains 0·01 mg of cadmium (as Cd).

A-5.3 Procedure — Pipette 10 ml aliquot of the solution prepared in **A-2.2.1** or **A-2.2.2** into a 400-ml beaker, dilute with water to about 40 ml and make neutral to litmus with sodium hydroxide solution.

A-5.3.1 Acidify with 2·5 ml of dilute hydrochloric acid and transfer to a 100-ml separating funnel, making the bulk up to approximately 50 ml with water. Extract with 10 ml of dithizone solution. The extract should remain green in colour, otherwise repeat the extraction with small quantities of dithizone until the carbon tetrachloride layer contains excess dithizone as indicated by its bluish-purple colour; two or three extractions are usually sufficient. Reject the dithizone extract.

A-5.3.2 To the aqueous solution contained in the separating funnel, add the whole of sodium potassium tartrate solution prepared in **A-5.2.5** and extract three times with 5-ml portions of dithizone solution, running the extracts into a clean 100-ml separating funnel. Extract once more with 5 ml of dithizone solution, running this extract into another separating funnel and confirm that the extraction of cadmium is complete by washing this extract twice with 2 ml each of sodium hydroxide solution (*see* Note). Separate and add the carbon tetrachloride layers to the first separating funnel. Extract the aqueous layer with 5 ml of carbon tetrachloride and add this to the combined extracts. Reject the aqueous layer.

NOTE — When the extraction is complete, washing with sodium hydroxide should give colourless carbon tetrachloride layer. If the carbon tetrachloride layer is pink, further extractions are necessary.

A-5.3.3 Shake the combined dithizone extracts with half its volume of sodium hydroxide solution and separate the dithizone extract. Repeat the treatment with sodium hydroxide solution, separate and finally wash the dithizone extract once with water. Run the dithizone extract into a 25-ml volumetric flask and dilute to the mark with carbon tetrachloride.

A-5.3.4 Measure the optical density of the solution at 27 ± 2°C at the wave length of 510 nm and determine the cadmium content in the material by referring to a previously prepared calibration graph (*see* **A-5.3.5**) showing micrograms of cadmium in relation to absorptiometer reading.

NOTE — If the optical density of the test solution indicates more than 10 µg of cadmium, repeat the optical density determination with a smaller volume of the solution diluted with carbon tetrachloride.

A-5.3.5 *Preparation of Calibration Graph* — Pipette 1 ml of standard cadmium solution into a 400-ml beaker and proceed as in **A-5.3** to **A-5.3.3.** Then transfer 0,1·0, 2·0, 3·0, 4·0, 5·0, 6·0, 7·0, 8·0 and 9·0 ml respectively of aliquots to ten 100-ml beakers, and add carbon tetrachloride to make up the volume to 10 ml to produce solutions having optical densities corresponding to 0, 1, 2, 3, 4, 5, 6, 7, 8 and 9 µg of cadmium content. Measure optical densities of these solutions and plot a graph of readings against cadmium content from 0 to 10 µg.

A-5.4 Calculation

$$\text{Cadmium (as Cd), percent by mass} = \frac{0{\cdot}000\,1\ M_1}{M}$$

where

M_1 = mass in micrograms of cadmium in the aliquot as read from the graph, and

M = mass in g of the material in the aliquot taken for the test.

A-6. DETERMINATION OF MERCURY

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 100-ml capacity (*see* IS : 4161-1967*).

A-6.2 Reagents

A-6.2.1 *Sodium Cyanide* — solid.

A-6.2.2 *Sodium Hydroxide* — solid (*see* IS : 376-1969†).

A-6.2.3 *Alkaline Stannous Chloride Solution* — Dissolve 1·2 g of stannous chloride crystals in 20 ml of water disregarding any precipitate formed by hydrolysis and pour whilst stirring into 20 ml of 20 percent (*m/v*) solution of sodium hydroxide. Prepare as required, filtering before use.

A-6.3 Procedure

A-6.3.1 *Zinc Oxide and Zinc Cyanide* — Dissolve 7 g of zinc oxide or 10 g of zinc cyanide in 50 ml of water containing 15 g of sodium cyanide and 5 g of sodium hydroxide. Cool, dilute to 100 ml and filter 50 ml through a filter paper (Whatman No. 40 is suitable) into a Nessler cylinder. Add 1 ml of alkaline stannous chloride solution and mix thoroughly. Carry out a blank determination on 15 g of sodium cyanide and 5 g of sodium hydroxide, and compare the turbidity, if any, developed with the sample in relation to that in the blank test.

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*Specification for Nessler cylinders.

†Specification for sodium hydroxide, analytical reagent (*first revision*).

A-6.3.2 *Zinc Sulphate and Zinc Chloride* — Dissolve 20 g of the material in 50 ml of water and add 15 g of sodium cyanide and 5 g of sodium hydroxide dissolved in 50 ml of water. Cool and filter 50 ml through a filter paper (Whatman No. 40 is suitable) into a Nessler cylinder and continue as in **A-6.3.1**.

A-6.3.3 The material shall be taken as having not exceeded the limits for mercury prescribed in Table 1 if the turbidity, if any, produced with the sample is not greater than that in the blank.

A-7. DETERMINATION OF COPPER

A-7.0 General — Copper is determined by measuring the optical density of copper-oxalyl-dihydrazide complex in a photoelectric absorptiometer at the wave length of 540 nm.

A-7.1 Apparatus

A-7.1.1 *Photoelectric Absorptiometer*

A-7.2 Reagents

A-7.2.1 *Hydrogen Peroxide* — 100 volumes.

A-7.2.2 *Citric Acid Solution* — Dissolve 50 g of citric acid (monohydrate) in 100 ml of water.

A-7.2.3 *Ammonium Hydroxide Solution* — 20 percent (*m/m*).

A-7.2.4 *Acetaldehyde Solution* — 40 percent (*v/v*) in methyl alcohol.

NOTE — As the boiling point of acetaldehyde is 21°C, mix the two liquids carefully in the cold.

A-7.2.5 *Oxalyldihydrazide Solution* — Dissolve 0·5 g of oxalyldihydrazide in 200 ml of water, heating slightly.

A-7.2.6 *Standard Copper Solution* — Dissolve 0·100 g of electrolytic copper foil in 20 ml of concentrated hydrochloric acid and 5 ml of hydrogen peroxide. After dissolution decompose the excess of hydrogen peroxide by boiling. Cool, transfer quantitatively to a 1 000-ml volumetric flask. Dilute with water up to the mark. Transfer 25 ml of this solution to a 250-ml volumetric flask, dilute with water up to the mark. One millilitre of this solution contains 0·01 mg of copper (as Cu).

A-7.3 Procedure — Pipette 50 ml of the solution prepared in **A-2.2.1** or **A-2.2.2** into a 200-ml beaker. Oxidize by adding a few drops of hydrogen peroxide and then evaporate to a syrupy consistency, cool and dilute with about 50 ml of water. Heat slightly to redissolve any residue, cool and transfer to a 100-ml volumetric flask filtering, if necessary; dilute to the mark and mix.

A-7.3.1 Transfer 10 ml aliquot to a 50-ml volumetric flask and add in turn 2 ml of citric acid solution and 5·5 ml of ammonium hydroxide solution and cool. Next add 20 ml of a mixture of 1 volume of acetaldehyde solution and 1 volume of oxalyldihydrazide solution that has been allowed to stand for 2 hours and filtered, if necessary. Swirl gently and dilute to the mark with water and mix.

A-7.3.2 Measure the optical density of the solution at 27 ± 2°C at the wave length of 540 nm and determine the copper content in the material by referring to a previously prepared calibration graph (*see* **A-7.3.3**) showing mg of copper in relation to the absorptiometer reading.

A-7.3.3 *Preparation of Calibration Graph* — Transfer into seven 50-ml volumetric flasks 0, 1, 2, 3, 4, 5 and 6 ml respectively of standard copper solution and add to each in turn 2 ml of citric acid solution and 5·5 ml of ammonium hydroxide solution. Cool and continue as in **A-7.3.1** to produce solutions having optical densities corresponding to 0·0, 0·01, 0·02, 0·03, 0·04, 0·05 and 0·06 mg respectively of copper content. Measure optical densities of these solutions and plot a graph of readings against copper content from 0 to 0·06 mg.

A-7.3.4 *Calculation*

$$\text{Copper content, percent by mass} = \frac{0{\cdot}1\, M_1}{M}$$

where

M_1 = mass in mg of copper in the aliquot as read from the graph, and

M = mass in g of the material in the aliquot taken for the test.

A-8. DETERMINATION OF IRON

A-8.0 General — Iron is determined by matching the colour produced by butanolic potassium thiocyanate solution with the colour produced in the standard iron solution.

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — 100-ml capacity (*see* IS : 4161-1967*).

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*Specification for Nessler cylinders.

A-8.2 Reagents

A-8.2.1 *Ammonium Persulphate*

A-8.2.2 *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water, transfer quantitatively to a 100-ml volumetric flask and make to the mark with *n*-butanol. Shake vigorously until the solution becomes clear.

A-8.2.3 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$FeSO_4.(NH)_2SO_4.6H_2O$] in about 100 ml of water containing 10 ml of dilute sulphuric acid [10 percent (*v/v*)], transfer to a 1 000-ml volumetric flask and dilute with water to 1 000 ml. Just before use, dilute 50 ml of the solution to 500 ml. One millilitre of this diluted solution contains 0·01 mg of iron (as Fe).

A-8.3 Procedure

A-8.3.1 *Zinc Oxide and Zinc Chloride* — Pipette 50 ml of the solution prepared in **A-2.2.1** into a 100-ml Nessler cylinder and add 30 mg of ammonium persulphate. Add 15 ml of butanolic potassium thiocyanate solution and shake vigorously for 30 seconds. Allow the two layers to separate. Simultaneously, carry out a control test in another Nessler cylinder using 7 ml of standard iron solution for zinc oxide and 2 ml for zinc chloride in place of the material, diluting to the 50-ml mark with water and adding the same quantities of the reagents as with the material. Compare the colour produced in the cylinders.

A-8.3.1.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-8.3.2 *Zinc Sulphate and Zinc Cyanide* — Pipette 50 ml of the solution prepared in **A-2.2.1** or **A-2.2.2** into a 100-ml volumetric flask, and dilute with water to the mark. Pipette 10 ml of this solution into a 100-ml Nessler cylinder, dilute to the 50-ml mark with water, add 30 mg of ammonium persulphate and continue as in **A-8.3.1**, carrying out control test using the following quantities of standard iron solution:

a) For zinc sulphate heptahydrate	2 ml
b) For zinc sulphate monohydrate	3 ml
c) For zinc cyanide	5 ml

A-9. DETERMINATION OF INSOLUBLE MATTER IN ZINC OXIDE

A-9.1 Reagent

A-9.1.1 *Sodium Cyanide Solution* — 10 percent (*m/v*). Filter before use.

A-9.2 Procedure — Weigh accurately about 20 g of the material, and dissolve in 500 ml of sodium cyanide solution by gentle warming and stirring for one hour. Filter through a tared sintered glass crucible (G No. 4), wash the residue with two 10-ml portions of warm sodium cyanide solution and then thoroughly with hot water. Dry at 110°C to constant mass.

A-9.3 Calculation

$$\text{Insoluble matter, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g of the residue, and
M = mass in g of the material taken for the test.

A-10. DETERMINATION OF INSOLUBLE MATTER IN ZINC SULPHATE

A-10.1 Reagent

A-10.1.1 *Dilute Sulphuric Acid* — approximately 3·5 N (10 percent *v/v*).

A-10.2 Procedure — Weigh accurately about 25 g of the material and add to 125 ml of water containing 1 ml of the dilute sulphuric acid. Heat to boiling, cool and filter through a tared sintered glass crucible (G No. 4). Wash the residue thoroughly with hot water and dry at 110°C to constant mass.

A-10.3 Calculation

$$\text{Insoluble matter, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g of the residue, and
M = mass in g of the material taken for the test.

APPENDIX B

(*Clauses* 3.1 *and* C-1.4)

SAFETY PRECAUTIONS FOR HANDLING ZINC CYANIDE AND OTHER RELATED INFORMATION

B-1. SAFETY PRECAUTIONS

B-1.1 As cyanides are highly poisonous, they shall never be touched with unprotected hands. Gloves shall be worn during sampling operations.

B-1.2 Solutions containing cyanide shall never be pipetted by mouth suction. A suitable safety pipette shall be used.

B-1.3 On account of the highly poisonous character of hydrogen cyanide all operations involving the decomposition of cyanide with acid shall be conducted in well-ventilated fume cupboard.

B-2. HYDROCYANIC ACID AND CYANIDE POISONING

B-2.1 Symptoms — The symptoms are giddiness, staggering and insensibility accompanied by panting respiration, and followed by profound collapse with convulsions. The action is extremely rapid.

B-2.2 First Aid

B-2.2.1 Remove the patient from cause of trouble, for example, fumes, etc, and take him to fresh air. Make the patient lie down, keep him warm and do not allow him to move more than necessary.

B-2.2.2 If breathing has ceased, apply artificial respiration.

B-2.2.3 Administer amyl nitrite. This is purchased in the form of small ampoules and one of these is broken and held under the nose so that the patient will inhale the vapour. It should be administered for 15 to 30 seconds every 2 to 3 minutes.

B-2.2.4 If available, administer oxygen through a face mask.

B-2.3 Medical Treatment

B-2.3.1 One method of treatment consists of injection into the bloodstream of sodium nitrite and sodium thiosulphate and should be carried out only by a qualified medical practitioner.

Details are as follows:

a) Intravenous injection of 0·3 g of sodium nitrite dissolved in 10 ml of sterile distilled water. This should be given slowly at the rate of 2·5 ml to 5 ml per minute.

b) Immediately following this and through the same needle, an intravenous injection of 25 g of sodium thiosulphate dissolved in 50-ml of sterile distilled water is given at the same rate. Leakage of material outside the vein should be avoided.

B-2.3.2 A temporary improvement is not a criterion of recovery. If the symptoms persist or recur after an hour, a second injection of the two substances should be given. It is suggested that shops using cyanide should keep a supply of these two substances in ampoule form and two sterilized syringes, one with a total capacity of 10 ml and the second with a total capacity of 50 ml, together with particulars of the treatment (as above).

B-2.3.3 Since it is unlikely that the average general practitioner or hospital would have such materials ready for use, should the patient be sent to hospital these materials should accompany him in the ambulance, and during the journey first-aid procedures already described continued.

B-2.3.4 Some patients may respond to first-aid treatment alone, but in many cases it will be advisable, if not necessary, to give the intravenous injection. This should be done as soon as possible, and in any case it is desirable that it should be administered within 15 minutes. It is, therefore, essential that if medical help cannot immediately be obtained, the patient should be conveyed without delay to the nearest hospital.

APPENDIX C

(*Clause* 5.1)

SAMPLING OF ZINC OXIDE AND ZINC SALTS FOR ELECTROPLATING

C-1. GENERAL REQUIREMENTS OF SAMPLING

C-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

C-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

C-1.2 The sampling instrument shall be clean and dry.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.4 Precautions shall be taken in handling zinc cyanide (*see* **3.1** and Appendix **B**).

C-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

C-1.6 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

C-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.8 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, and the date of sampling.

C-2. SCALE OF SAMPLING

C-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

C-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FROM LOTS OF DIFFERENT SIZES

NUMBER OF CONTAINERS IN THE LOT N (1)	SAMPLE SIZE n (2)
3 to 15	3
16 ,, 40	4
41 ,, 110	5
111 ,, 180	6
181 ,, 300	7
301 ,, 500	8
501 ,, 800	9
801 and above	10

C-2.3 The containers shall be selected at random. In order to ensure randomness of selection, a random number table shall be used. For guidance and use of random number tables, IS : 4905-1968* may be referred. In the absence of random number tables, the following procedure may be adopted:

> Starting from any container in the lot, count them as 1, 2, 3,..., etc, up to r and so on, where r is the integral part of N/n (N being the number of containers in the lot and n the sample size). Every rth container thus counted shall be withdrawn so as to constitute the required sample size.

C-3. INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

C-3.1 From each of the containers selected according to **C-2.2**, a representative portion of the material sufficient for carrying out the tests shall be drawn and these shall constitute the individual samples.

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*Methods for random sampling.

C-3.2 From each of the individual samples, a small but equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample.

C-3.3 The material in the individual and composite samples shall be transferred to separate bottles and labelled with full identification particulars of the samples.

C-4. NUMBER OF TESTS

C-4.1 Tests for the determination of zinc, zinc oxide and cyanide contents shall be carried out on each of the individual samples.

C-4.2 Tests for the remaining characteristics shall be done on the composite sample.

C-5. CRITERIA FOR CONFORMITY

C-5.1 For Individual Samples — For zinc, zinc oxide and cyanide contents respectively, as prescribed, the results shall be noted and their mean (X) and the range (R), being the difference between the maximum and the minimum of test results, shall be computed. For declaring the conformity of the lot in respect of these characteristics ($X - 0{\cdot}6\ R$) shall be greater than or equal to the minimum specified in Table 1.

C-5.2 For Composite Sample — For declaring the conformity of the lot to the requirements of all other characteristics, the test results of the composite sample shall satisfy the relevant requirements specified in Table 1.

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